

# Image Cover Sheet

**CLASSIFICATION**

UNCLASSIFIED

**SYSTEM NUMBER**

511887



**TITLE**

Mineralogical Characterization of Airborne Industrial Pollutants

**System Number:**

**Patron Number:**

**Requester:**

**Notes:** Paper #13 contained in Parent sysnum #511874

**DSIS Use only:**

**Deliver to:** CL



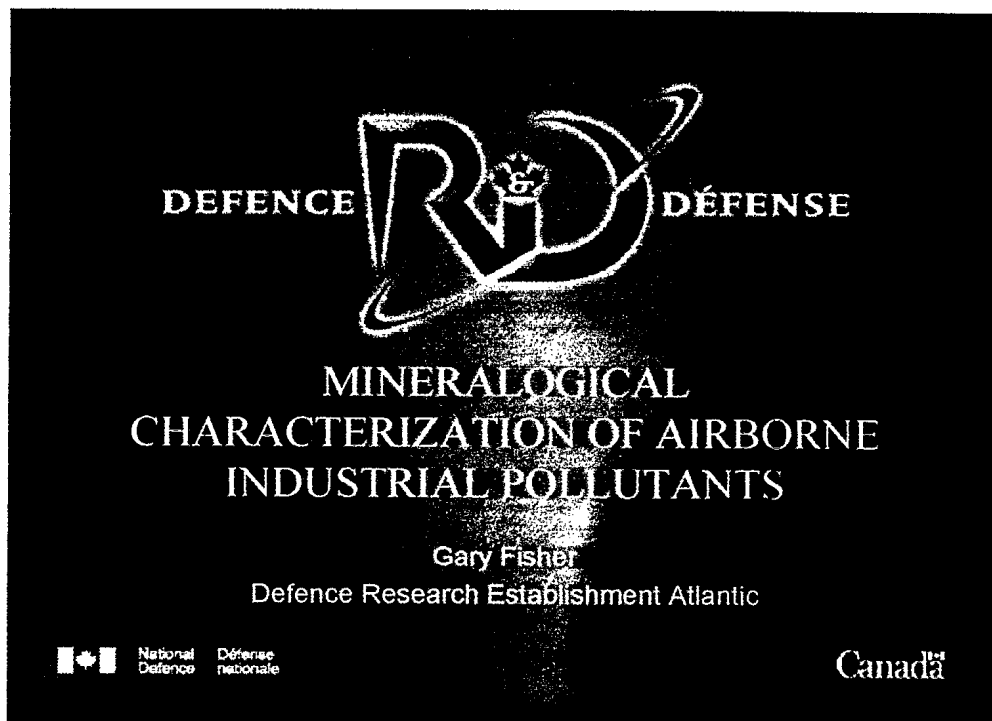
# **Mineralogical Characterization of Airborne Industrial Pollutants**

by Gary Fisher

**Defence Research Establishment Atlantic, Dockyard Laboratory (Atlantic)  
P.O. Box 99000, Stn Forces, Halifax, Nova Scotia, Canada B3K 5X5**

## **ABSTRACT**

Increased awareness of environmental and health-related issues has led to heightened concern about air quality in industrial and office settings. Often the airborne pollutants that cause personnel complaints of eye, nose and throat irritation are inorganic particulates released by industrial processes, dirty or inadequate ventilation systems, or poor housecleaning practices. Identification of these particulates and their source is a necessary first step to improving industrial air quality. However, due to the small quantities of airborne material available in even the dirtiest of samples, standard analytical techniques are not practical for such identification. This paper describes the use of transmission polarized light microscopy, both alone and in conjunction with scanning electron microscopy/energy dispersive x-ray microanalysis, as a means of identifying airborne inorganic particulates at the picogram level.



## OUTLINE

- History
- Analysis options
  - X-ray diffraction
  - Scanning electron microscopy/x-ray microanalysis
  - Transmission polarized light microscopy
- Mineralogical characterization
- Results

## HISTORY

- Industry/government respond to health & safety and political issues of the day
- 1970's - reduction in use of toxic industrial materials
  - asbestos, lead, PCBs
- 1980's - global environment
  - recycling, eco-products, spill prevention
- 1990's - personal working environment
  - ventilation issues, fugitive emissions, allergens, individual sensitivities



Defence Research Establishment Atlantic (DREA)

## HISTORY

- Increase in general health complaints
  - eye/nose/throat irritations
  - headaches
  - nausea/drowsiness/general malaise
  - transitory



Defence Research Establishment Atlantic (DREA)

## HISTORY

- Air quality issues
  - fumes, fugitive emissions, particulates
- Airborne particulate content is recognized as a health & safety issue
  - 50  $\mu\text{g}/\text{m}^3$  particles < 10  $\mu\text{m}$  proposed limit



Defence Research Establishment Atlantic (DREA)

## ANALYSIS OPTIONS

- Require a technique able to identify airborne particulates
  - Natural:
    - minerals, pollen, plant fibers
  - Synthetic:
    - fibers, soot (fly ash), minerals



Defence Research Establishment Atlantic (DREA)

## ANALYSIS OPTIONS

- X-ray diffraction
  - identify crystallographic species
  - problems with polymers and natural materials
  - insensitive: 5% detection limit
- Microscopic technique
  - Scanning electron microscopy/energy dispersive x-ray microanalysis (SEM/EDX)
  - Transmission polarized light microscopy



Defence Research Establishment Atlantic (DREA)

## SEM/EDX

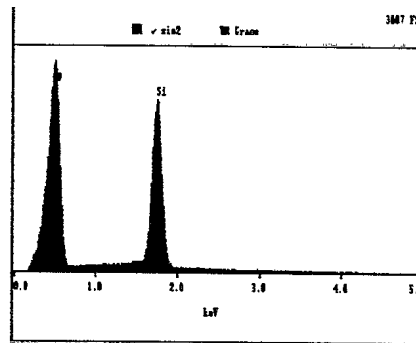
- Superior resolving power and depth of focus
- Non-conductive samples problematic
- No colour



Defence Research Establishment Atlantic (DREA)

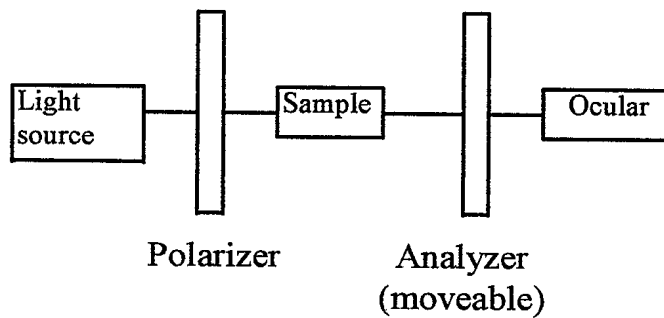
## SEM/EDX

- X-ray emission enables chemical analysis
- Elemental analysis versus identification of species
  - quartz indistinguishable from glass



Defence Research Establishment Atlantic (DREA)

## TRANSMISSION POLARIZED LIGHT MICROSCOPY



Defence Research Establishment Atlantic (DREA)



## TRANSMISSION POLARIZED LIGHT MICROSCOPY

### Sample Preparation

- Examined using stereomicroscope to select particle types and/or representative sample
- Sieved and/or crushed
- Sample mounted in refractive index fluid,  $n_D = 1.550$
- Examined at low magnification (10X objective) to determine particle types



Defence Research Establishment Atlantic (DREA)

## POLARIZED VS. UNPOLARIZED



Unpolarized



Polarized

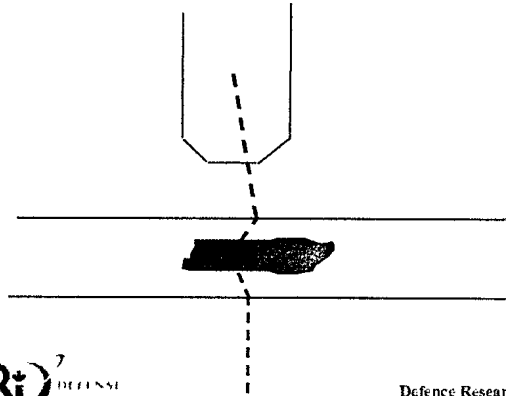


Crossed  
polars



Defence Research Establishment Atlantic (DREA)

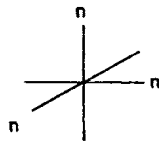
## TRANSMITTANCE PLM



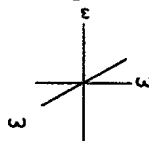
Defence Research Establishment Atlantic (DREA)

## OPTICAL PROPERTIES

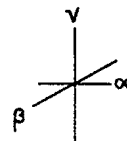
- $n = C_v/C_m$
- Optical isotropy - 1 refractive index
- Optical anisotropy - multiple refractive indices



Isotropic  
cubic



Anisotropic  
tetragonal  
hexagonal



Anisotropic  
orthorhombic  
monoclinic  
triclinic



Defence Research Establishment Atlantic (DREA)

## POLARIZED VS. UNPOLARIZED



Unpolarized



Polarized



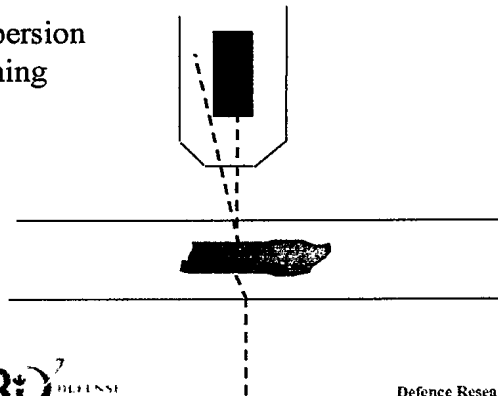
Crossed  
polars



Defence Research Establishment Atlantic (DREA)

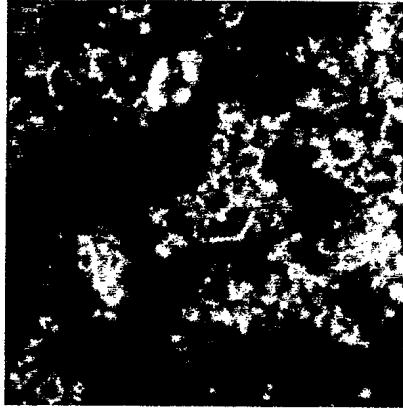
## DETERMINATION OF REFRACTIVE INDEX

Dispersion  
staining



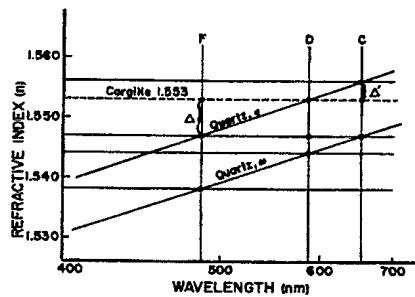
Defence Research Establishment Atlantic (DREA)

# DETERMINATION OF REFRACTIVE INDEX



Defence Research Establishment Atlantic (DREA)

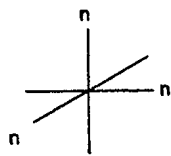
# DETERMINATION OF REFRACTIVE INDEX



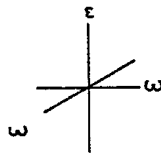
Defence Research Establishment Atlantic (DREA)

## CHARACTERIZATION OF NON-CUBIC CRYSTALS

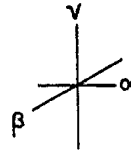
Anisotropic crystals show 1 or 2 refractive indices, regardless of orientation



Isotropic  
cubic



Anisotropic  
tetragonal  
hexagonal

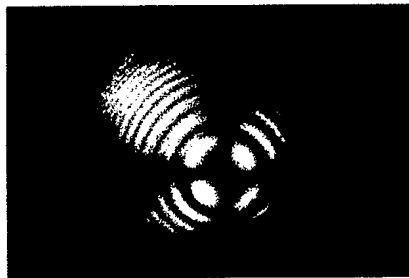


Anisotropic  
orthorhombic  
monoclinic  
triclinic



Defence Research Establishment Atlantic (DREA)

## CHARACTERIZATION OF NON-CUBIC CRYSTALS



UNIAXIAL



BIAXIAL



Defence Research Establishment Atlantic (DREA)

## CHARACTERIZATION OF NON-CUBIC CRYSTALS

### BIREFRINGENCE

- Light exiting one axis is "retarded" relative to the other
- Yields interference colors

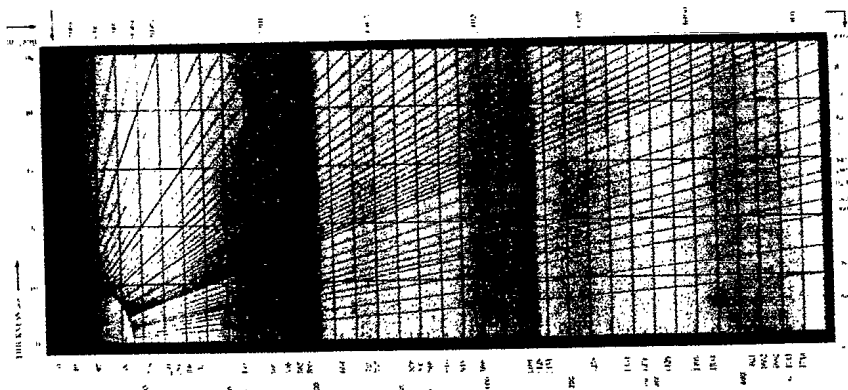
$$B = \frac{\text{retardation (nm)}}{\text{thickness (nm)}}$$



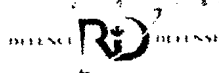
Defence Research Establishment Atlantic (DREA)



## CHARACTERIZATION OF NON-CUBIC CRYSTALS



Defence Research Establishment Atlantic (DREA)



## SUMMARY

### ISOTROPIC MINERALS

- Cubic or amorphous
- Measure  $n$  by dispersion staining
- Color
- X-ray microanalysis can aid identification

### ANISOTROPIC MINERALS

- Conoscopic imaging to determine uniaxial/biaxial
- Measure birefringence from Michel-Levy chart
- Color/morphology
- X-ray microanalysis can aid identification



Defence Research Establishment Atlantic (DREA)

## MINERAL PROPERTIES

<u>MINERAL</u>	<u>B</u>	<u>n</u>	<u>SYSTEM</u>
Halite	NA	1.544	Cubic
Corundum	0.007	$\omega$ - 1.772 $\varepsilon$ - 1.763	Rhomb.
Quartz	0.009	$\omega$ - 1.544 $\varepsilon$ - 1.553	Rhomb.
Calcite	0.190	$\omega$ - 1.740 $\varepsilon$ - 1.550	Rhomb.
Gypsum	0.010	$\alpha$ - 1.521 $\beta$ - 1.526 $\gamma$ - 1.531	Monoclinic



Defence Research Establishment Atlantic (DREA)

## RESULTS

### Background Mineral Content

- quartz, sodium chloride, gypsum ( $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ )

### Problem Minerals

- quartz, iron oxides, rutile ( $\text{TiO}_2$ ), ground glass

### Other Materials

- fibers, hair (human and rodent), pollen/seed hairs



Defence Research Establishment Atlantic (DREA)

The Defence Research  
and Development Branch  
provides Science and  
Technology leadership  
in the advancement and  
maintenance of Canada's  
defence capabilities.

